

From: Kelley Chase/R3/USEPA/US
Sent: 6/12/2012 3:35:41 PM

To: Richard Fetzer/R3/USEPA/US@EPA
CC:
Subject: Fw: EXTERNAL: Re: Verification/Completeness Check for File 1202013 FINAL PART 2 of 3 R33907 02 11 12 1537.pdf Posted Feb 13

This is a follow-up to last e-mail.

- Initial comments by [redacted] on 2/16
- Response from CC on 2/27
- Follow-up [redacted] on 3/1
- Response CC on 3/5
- Response [redacted] on 3/5
- There are two more e-mails on 3/5 related to this review - Stevie Wilding response to [redacted] and [redacted] response indicating the final resolution.

----- Forwarded by Kelley Chase/R3/USEPA/US on 06/12/2012 03:30 PM -----

From: [redacted] Ex. 4 - CBI
To: Cynthia Caporale/ESC/R3/USEPA/US@EPA
Cc: [redacted] Ex. 4 - CBI, Gary Newhart/CI/USEPA/US@EPA, John Gilbert/CI/USEPA/US@EPA, Kelley Chase/R3/USEPA/US@EPA, Robin Costas/ESC/R3/USEPA/US@EPA, Sella Burchette/ERT/R2/USEPA/US@EPA, Stevie Wilding/ESC/R3/USEPA/US
Date: 03/05/2012 12:24 PM
Subject: RE: EXTERNAL: Re: Verification/Completeness Check for File 1202013 FINAL PART 2 of 3 R33907 02 11 12 1537.pdf Posted Feb 13

Cynthia,

I am looking at the lab report for 1201013 FINAL DASR33907 02 11 12 1538 and for Batch BA23102-BS-1 the % recovery for 2-methoxyethanol is 0%. Since this is done at the mid-level spike of 60 ug/L, the Quantitation Limit cannot be raised to 60 ug/L. The BS-2 recovery for this batch is 66%. Is there an error in reporting on the lab report? I am seeing 0%/66% for the LCS recoveries.

[redacted] Ex. 4 - CBI

From: Cynthia Caporale [mailto:Caporale.Cynthia@epamail.epa.gov]
Sent: Monday, March 05, 2012 12:09 PM
To: [redacted] Ex. 4 - CBI
Cc: [redacted] Ex. 4 - CBI, Gary Newhart; John Gilbert; Kelley Chase; Robin Costas; Sella Burchette; Stevie Wilding
Subject: RE: EXTERNAL: Re: Verification/Completeness Check for File 1202013 FINAL PART 2 of 3 R33907 02 11 12 1537.pdf Posted Feb 13

[redacted] Ex. 4 - CBI and Kelley,

Below after each bullet are our comments in black.

Based on the response from the R3 Lab dated 2/27/12, the data included in File 1201013 FINAL PART 2 of 3 R33907 02 11 12 11537.pdf and included in the Verification/Completeness Check issued on 2/15/12 (document SERAS-001-DSR-021612_Dimock_3), the following qualifications are recommended:

☐ For the SVOC analysis (item #1), the reporting level for FB01 should be elevated to the mid-level spike concentration. It cannot be ascertained from the information available to the reviewer if this level is 20 or 40 ug/L.

Response: The mid-level spike concentration is 60 ug/L.

□ For the SVOC analysis (item #2), many of the low-level contaminants found in the method blank were also found in the field blanks. All of these concentrations were under the reporting limit; thereby making it difficult to determine whether the data were qualified based on the method and/or field blank. It is recommended that the National Functional Guidelines are used to qualify data as below. As an example in batch BA22504, prepped on 1/25/12, the method blank contained 0.408 ug/L of bis-2-thylhexylphthalate and 0.966 ug/L of di-n-butylphthalate. The field blank contained 0.358 ug/L of bis-2-ethylhexylphthalate and 0.668 ug/L of di-n-butylphthalate. Essentially the field blank results are less than the method blank results and should be qualified “U” with a RL of 5 ug/L. This is consistent through many of the batches and would qualify the phthalate results as “U” in several samples.

Response: Following NFG protocols for blank contamination is a project-level decision. Typically, for R3 validation reports, we retain the "B" qualifiers for the risk assessors (at least for now); however, if the NFG approach is more appropriate for this project then we are supportive of that decision.

Table 31. Blank Actions for Semivolatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field	Detects	Not detected	No qualification
	< CRQL*	< CRQL*	Report CRQL value with a U
	> CRQL*	< CRQL*	Use professional judgment
			Report CRQL value with a U
	> CRQL*	> CRQL* and <blank concentration	Report the blank concentration for the sample with R
		> CRQL* and > blank concentration	Use professional judgment
	= CRQL*	< CRQL*	Report CRQL with a U
		> CRQL*	Use professional judgment
	Gross contamination	Detects	Qualify results as unusable R
	TIC > 10 µg/L (for aqueous blanks) TIC > 330 µg/kg (for nonaqueous blanks)	Detects	Use professional judgment

* 5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples.

□ For SVOC analysis (item #3), the quantitation limit cannot be elevated to the mid-level spike since the LCS theoretical value is 60 ug/L, which is at mid-level. The sample results for 2-methoxyethanol that are associated with this LCS (Batch B23102) should be qualified “R” unusable.

Response: The mid-level spike (called BS2 or LCS at 60 ug/L) was acceptable so the quantitation limit could be raised, as per our initial response.

□ For VOC analysis, further clarification is needed. For Freon 113, methylacetate, methylcyclohexane, cyclohexane and MTBE, there are no accuracy data reported for the LCS or the MS. In your response, you state that not all LCS compounds are reported in the lab report due to issues with the Element system . It also states that a LCS is not available for these compounds. These two statements contradict each other. Did you mean that these compounds are in the MS but aren’t reported due to issues with the LIMS? If these compounds are present in the MS but not in the LCS and the recoveries are within the QC limits except for the two noted that were high, then no qualification of the data is needed.

Response: Yes, as per your last statement above, these compounds are present in the MS but not in the LCS and the recoveries are within the QC limits except for the two noted that were high and no qualification of data is needed.

□ For the acetone result for sample HW14-P flagged as “K” (biased high), the lab qualifier can remain as a “K” but the result qualifier should be noted as “J” in Scribe since these qualifiers are going into two separate columns.

Response: We agree.

If you should have any questions please contact me or Robin Costas (410-305-2659).

Cynthia Caporale, Chief
OASQA Laboratory Branch
U.S. EPA Region III
Environmental Science Center
Fort Meade, MD
(410) 305-2732
Fax: (410) 305-3095

From:

Ex. 4 - CBI

To: Cynthia.Caporale/ESC/R3/USEPA/US@EPA, Kelley Chase/R3/USEPA/US@EPA
Cc:

Ex. 4 - CBI

, Gary Newhart/CI/USEPA/US@EPA, John Gilbert/CI/USEPA/US@EPA, Sella Burchette/ERT/R2/USEPA/US@EPA, Robin Costas/ESC/R3/USEPA/US@EPA, Stevie Wilding/ESC/R3/USEPA/US
Date: 03/01/2012 10:24 AM
Subject: RE: EXTERNAL: Re: Verification/Completeness Check for File 1202013 FINAL PART 2 of 3 R33907 02 11 12 1537.pdf Posted Feb 13

Cynthia and Kelley,

Based on the response from the R3 Lab dated 2/27/12, the data included in File 1201013 FINAL PART 2 of 3 R33907 02 11 12 11537.pdf and included in the Verification/Completeness Check issued on 2/15/12 (document SERAS-001-DSR-021612_Dimock_3), the following qualifications are recommended:

- ☐ For the SVOC analysis (item #1), the reporting level for FB01 should be elevated to the mid-level spike concentration. It cannot be ascertained from the information available to the reviewer if this level is 20 or 40 ug/L.
- ☐ For the SVOC analysis (item #2), many of the low-level contaminants found in the method blank were also found in the field blanks. All of these concentrations were under the reporting limit; thereby making it difficult to determine whether the data were qualified based on the method and/or field blank. It is recommended that the National Functional Guidelines are used to qualify data as below. As an example in batch BA22504, prepped on 1/25/12, the method blank contained 0.408 ug/L of bis-2-thylhexylphthalate and 0.966 ug/L of di-n-butylphthalate. The field blank contained 0.358 ug/L of bis-2-ethylhexylphthalate and 0.668 ug/L of di-n-butylphthalate. Essentially the field blank results are less than the method blank results and should be qualified “U” with a RL of 5 ug/L. This is consistent through many of the batches and would qualify the phthalate results as “U” in several samples.

Table 31. Blank Actions for Semivolatiles Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field	Detects	Not detected	No qualification
	< CRQL*	< CRQL*	Report CRQL value with a U
	> CRQL*	< CRQL*	Use professional judgment
			Report CRQL value with a U
	> CRQL*	> CRQL* and <blank concentration	Report the blank concentration for the sample with a U
		> CRQL* and > blank concentration	Use professional judgment
	= CRQL*	< CRQL*	Report CRQL with a U
		> CRQL*	Use professional judgment
	Gross contamination	Detects	Qualify results as unusable R
	TIC > 10 µg/L (for aqueous blanks) TIC > 330 µg/kg (for nonaqueous blanks)	Detects	Use professional judgment

* 5x the CRQL for bis(2-ethylhexyl)phthalate for low-level non-aqueous and aqueous samples.

- ☐ For SVOC analysis (item #3), the quantitation limit cannot be elevated to the mid-level spike since the LCS theoretical value is

60 ug/L, which is at mid-level. The sample results for 2-methoxyethanol that are associated with this LCS (Batch B23102) should be qualified “R” unusable.

☐ For VOC analysis, further clarification is needed. For Freon 113, methylacetate, methylcyclohexane, cyclohexane and MTBE, there are no accuracy data reported for the LCS or the MS. In your response, you state that not all LCS compounds are reported in the lab report due to issues with the Element system . It also states that a LCS is not available for these compounds. These two statements contradict each other. Did you mean that these compounds are in the MS but aren’t reported due to issues with the LIMS? If these compounds are present in the MS but not in the LCS and the recoveries are within the QC limits except for the two noted that were high, then no qualification of the data is needed.

☐ For the acetone result for sample HW14-P flagged as “K” (biased high), the lab qualifier can remain as a “K” but the result qualifier should be noted as “J” in Scribe since these qualifiers are going into two separate columns.

If there are any questions or comments, please do not hesitate to call.

Ex. 4 - CBI

From: Cynthia Caporale [mailto:Caporale.Cynthia@epamail.epa.gov]
Sent: Monday, February 27, 2012 4:40 PM
To: Ex. 4 - CBI
Cc: Ex. 4 - CBI; Gary Newhart; John Gilbert; Kelley Chase; Sella Burchette; Robin Costas; Stevie Wilding
Subject: EXTERNAL: Re: Verification/Completeness Check for File 1202013 FINAL PART 2 of 3 R33907 02 11 12 1537.pdf Posted Feb 13

The report on the Dimock Verification/Completeness Check for file 1201013 FINAL Part 2 of 3 R33907 02 11 12 1537.pdf was reviewed and below are the responses for your consideration.

File 1201013 FINAL PART 2 of 3 R33907 02 11 12 1537.pdf

1. For SVOC analysis, the low level spike recovery for 2,4-dinitrophenol associated with sample FB-01 was 0%. It cannot be determined from the laboratory report if this low level spike is at the LOQ. Since this is a problematic compound, should the “UJ” be changed to unusable “R” for this sample?

Response: The low-level spike concentration for 2,4-dinitrophenol is at the LOQ (limit of quantitation) of 5 ug/L. The mid-level spike result did show acceptable recovery. Therefore, we don't recommend qualifying "R;" however, elevating the quantitation limit to the mid-level spike value might be more appropriate. (Note: This is why "UJ" qualifier is used - notes an issue with the normal quantitation limit but other quality control results were acceptable) For future reports related to this project we will elevate the QL; however, this is not our typical practice.

2. For SVOCs, it appears that flags were assigned to samples based on contaminants found in the corresponding method blanks; however, it appears that samples were not qualified based on contaminants in the corresponding field blanks. The Region needs to decide if this is or should be part of their validation process. For example when using the National Functional Guidelines for Data Review, the samples are first qualified on the basis of the method blank and then the field blank (and in the case of VOCs the trip blank also). This would eliminate most of the “J values reported (>MDL but <RL and the results raised to RL). As an example, the samples prepped on 1/29/12 are associated with B22901 and also FB-02 and FB-03. Many of the contaminants present in the blanks are very similar in concentration to the samples.

Response: These samples were qualified based on Field Blanks. Please provide more specific information on which analyte you feel is not qualified. (Was these qualifiers missing from the EDD?)

3. For SVOCs prepared on 1/31/12 in B23102, the 2-methoxyethanol recovery for LCS_BS1 was 0%. Since this recovery was 0%, should the samples that were non-detect be reported as unusable “R” instead of “UJ”?

Response: The mid-level spike result did show acceptable recovery. Therefore, as stated above, we don't recommend qualifying "R" however, the quantitation limit could be elevated to the mid-level spike value. For future reports related to this project we will elevate the QL; however, this is not our typical practice.

4. For VOC analysis, there doesn’t appear to be any precision and accuracy data for Freon 113, methylacetate, methyl cyclohexane or MTBE for the LCS or the MS. The Region needs to decide whether these results should be flagged as estimated “J” or a note placed in the case narrative stating that these data are not available for these compounds.

Response: The above note also applies to cyclohexane. Not all LCS compounds are reported in the lab report due to issues

with the Laboratory Information Management system; however, this information is available in the case files. We do not have an LCS for Freon 113, methyl acetate, MTBE, cyclohexane or methyl cyclohexane. These are part of the matrix spikes done for samples 1201013-14 and 1201013-33. Recoveries for all 5 compounds were within limits of 80-120%, except for sample 1201013-14 for cyclohexane (122%)and for sample 1201013-33 for methyl acetate (124%).

5. For the acetone result flagged as “K” on the report table and in the case narrative, should a “J” flag also be entered indicating that this result is an estimated value probably biased high?

Response: Notes and Definitions page does state that a result qualified with a "K" is estimated. At the project level any "L" or "K" qualifier can be changed to "J" as estimated. The "L" and "K" qualifiers are providing information on bias. We do not recommend using both the "J" and the "K" together.

6. It is assumed that all required instrument QC (RSD, %D, minimum response factors, etc.) specified by the method was run and was within the criteria listed in the EPA R3 SOPs since this information is not available in the laboratory report.

Response: This assumption is correct and future reports will include a statement in the narrative.

Overall, based on the above comments and response an impact to result values or qualifiers does not seem warranted.

If you should have any questions or need further discussion on the above response please feel free to contact me or Robin Costas at 410-305-2659.

Cynthia Caporale, Chief
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(410) 305-2732
Fax: (410) 305-3095

From: Ex. 4 - CBI
To: Kelley Chase/R3/USEPA/US@EPA, Cynthia Caporale/ESC/R3/USEPA/US@EPA
Cc: John Gilbert/CI/USEPA/US@EPA, Gary Newhart/CI/USEPA/US@EPA, Sella Burchette/ERT/R2/USEPA/US@EPA Ex. 4 - CBI
Date: 02/16/2012 02:40 PM
Subject: Verification/Completeness Check for File 12013 FINAL PART 2 of 3 R33907 02 11 12 1537.pdf Posted Feb 13

.....is attached.

Ex. 4 - CBI
Lockheed Martin
Scientific, Engineering, Response and Analytical Services (SERAS)

Ex. 4 - CBI
[attachment "SERAS-001-DSR-021612_3.docx" deleted by Cynthia Caporale/ESC/R3/USEPA/US]